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Optimizing Heat Treatment Parameters for 3rd Generation AHSS Using an Integrated Experimental-Computational Framework

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PACIFIC NORTHWEST NATIONAL LABORATORY
RICHLAND, WA, USA

2018 DOE VTO ANNUAL MERIT REVIEW
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PROJECT ID# MAT129

Overview

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Timeline

- Project Start Date: FY16
- Project End Date: FY19
- Percent complete: 60%

Budget

- Total project funding (FY16-17)
  - DOE : $1,599k
  - ASPPRC In-kind: $400k
- Funding for FY16: $400k
- Funding for FY17: $399k
- Funding for FY18: $500k
- Funding for FY19: $300k

Barriers

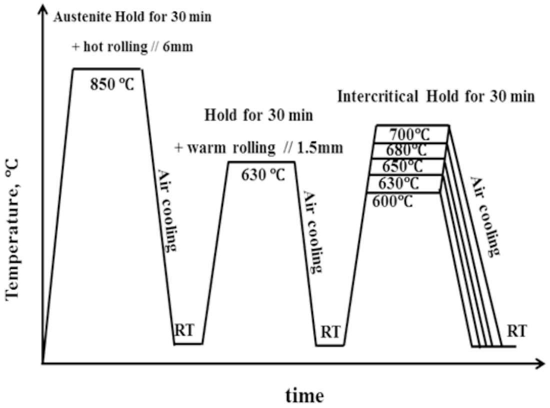
- The traditional heat treatment (HT) and characterization process make the development-to-deployment cycle of 3rd GEN Med-Mn AHSS very long
- Lack of fundamental and quantitative understanding between alloying content, HT parameters, microstructures and associated mechanical properties of Med-Mn AHSS

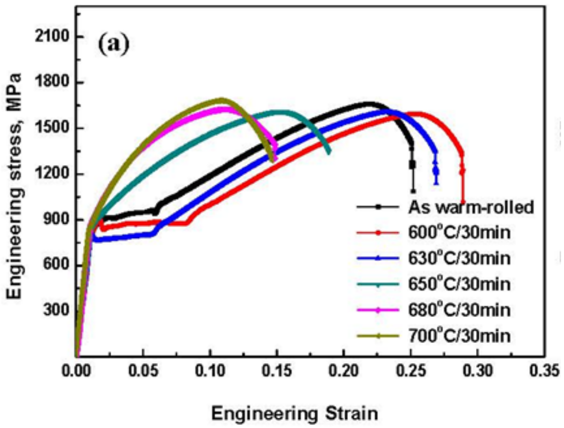
Partners

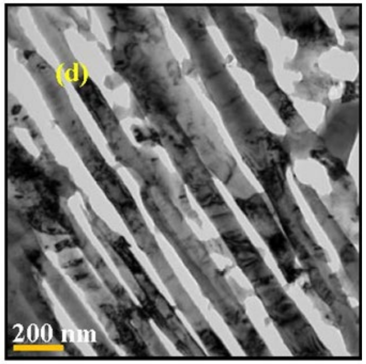
- Pacific Northwest National Lab (PNNL)
- Advanced Steel Processing and Products Research Center (ASPPRC), Colorado School of Mines (CSM)
- Advanced Photon Source (APS), Argonne National Lab (ANL)

Relevance/Objectives

- Development of an integrated in-situ & ex-situ experimental and numerical modeling framework for Med-Mn 3Gen AHSS to
  - Determine accurate thermodynamic parameters.
  - Create a framework for optimizing the inter-critical annealing parameters for a given Mn content that results in desired/improved strength and ductility.
  - Meet DOE VTO MYPP targets and goals.
  - Help steel makers and users to expedite the development-to-deployment cycle







Zhao, et al., *Materials* 2014, 7

Milestone

Date	Milestone or Go/No-go	Goal description	Status
9/30/2016	Milestone	Develop a high throughput HEXRD-based *in-situ* characterization process to obtain desired RA volume fraction and stability for 3rd GEN AHSS	Completed
9/30/2018	Milestone	A phase field modeling framework that can predict the effects of heat treatment parameters (i.e., IA temperature and time) on the phase volume fractions as well as C and Mn content in each phase	Completed
9/30/2019	Milestone	Validate phase field model prediction of solute partioning	On track

Approach

Measurement

- In-situ HEXRD IA heat treatment experiment
- Ex-situ heat treatment experiment and characterization
- In-situ HEXRD tensile testing

Results

- AVF variation due to time and temperature
- PLP variation due to time and temperature
- C & Mn partioning

Modeling

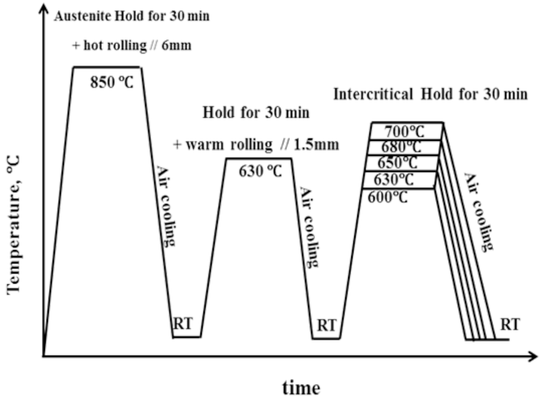
- Phase field modeling capability (DICTRA , MICRESS)
- Thermodynamic Parameters
- Microstructure-based finite element modeling
- Macroscopic tensile properties

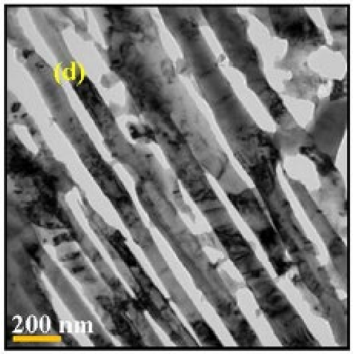
Optimized IA parameters

IA = Intercritical annealing AVF = Austenite volume fraction PLP = Phase lattice parameters

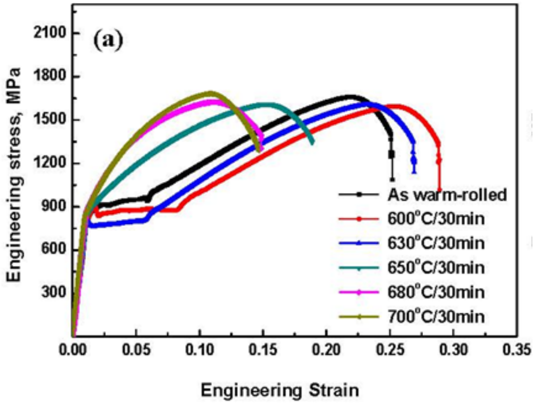
Medium Mn Steels & Heat Treatment Processes

- Med-Mn steel is an 3rd Gen AHSS which *can have* excellent combination of ductility and strength due to the austenite to martensite phase transformation during forming.
- Inter-critical annealing (IA) process after austenization is used to optimize the microstructure, esp. retained austenite volume fraction (RAVF).
- Both temperature and holding time affect the microstructure and RAVF leading to different strength and ductility values.
- Ex-situ characterization is usually utilized after various heat treatment processing with different IA parameters, which is costly and time consuming
- An in-situ characterization during heat treatment processing can provide great opportunities to obtain full information of phase transformation during the whole process.





Fe-7.9Mn-0.14Si-0.05Al-0.07C Steel



Accomplishments to Date

- High throughput *in-situ* HEXRD intercritical annealing (IA) heat treatment experiment has been developed & utilized for three Med-Mn steels : 5Mn, 7Mn and 10Mn.
- *Ex-situ* heat treatment and characterization (SEM, HEXRD) has been completed.
- ThermoCalc-Dictra (TCD) simulations for 5Mn steels haven been developed to study phase transformation during IA holding.
- **Continued exploration of Mn mobility** in ferrite/martensite in ~5Mn steel, including
  - determination of a mobility enhancement factor using the integrated experimental (ex-situ & in-situ) and numerical modeling(TCD) framework.
  - direct calculation from HEXRD measurements.
- **Continued development of the phase field microstructure modeling framework**
  - Modeling of microstructure evolution and partitioning over time
  - **Modeling the effect of heating rate on austenite growth and ferrite recrystallization**
- Non-incremental *in-situ* HEXRD tensile tests of 7Mn & 10Mn steels along different directions to better under the TRIP effect and the yield point elongation (Luders band behavior) have been conducted.
- Microstructure/mechanism-based phenomenological and crystal plasticity FE models have been developed with the consideration of martensitic transformation.
- Luders band and yield point elongation (YPE) behavior have been successfully captured by the above mentioned models.

Determination of Mn Mobility by TCD and in-situ HEXRD data

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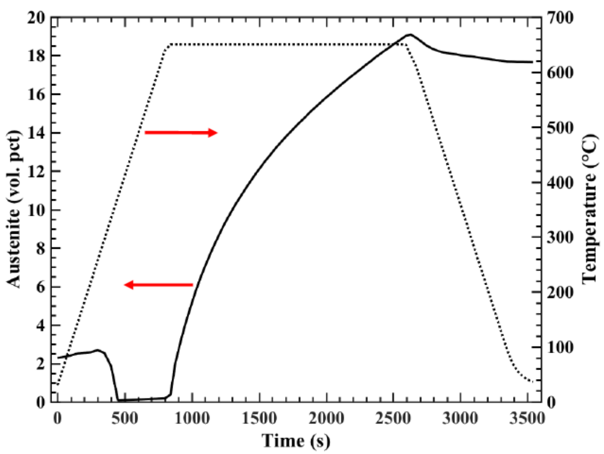
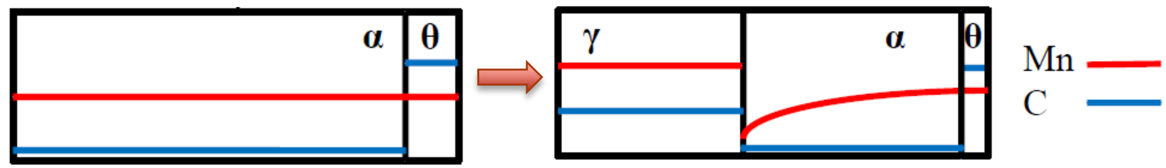
- Mn mobility was obtained with consideration of austenite decomposition and cementite formation during heating



In-situ HEXRD

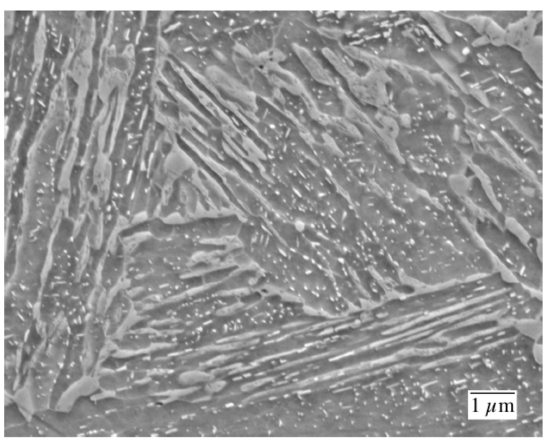


Initial DICTRA cell

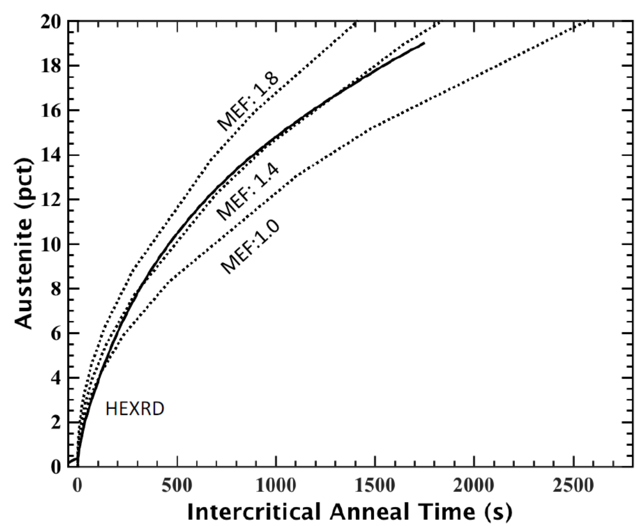
DICTRA cell upon IA

Model Parameters

Nominal Composition: 0.19C-4.39Mn
Phase Widths: γ-NA, α-203.9 nm, θ-6.09 nm (2.9 vol pct θ)
Phase Compositions(wt pct): α: 2.21×10^{-7}C-4.39Mn, θ: 6.73C-4.39Mn
IAT: 650 °C
Mobility Enhancement Factor: Mn in BCC



0.8°C/s HR, 650°C & 1000s IA

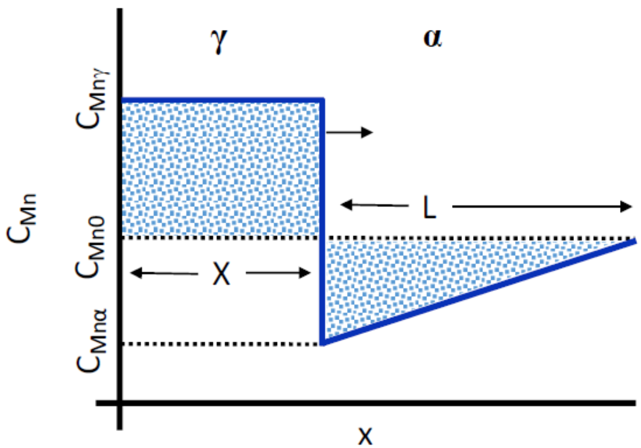


- MEF of 1.4 agrees with experiment
- Trajectories don't match well
- Mn diffusivity may decay due to recrystallization and recovery

Accomplishments to Date

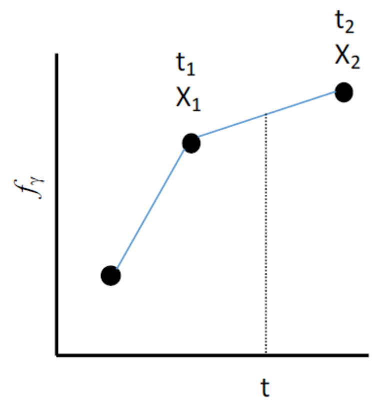
Direct calculation of Mn Diffusivity from HEXRD data

- Assumptions
  - Mn diffusion controlled interface growth
  - Austenite growth occurs at equilibrium composition
  - Infinitely fast C diffusion
  - Linear Mn gradient in ferrite
  - Same geometry as DICTRA cell w/o cementite (210nm α)



dx/dt : interface velocity
X : half width of austenite
C_{Mn0} : 0.0443
$C_{Mn\alpha}$: 0.0193
$C_{Mn\gamma}$: 0.0813

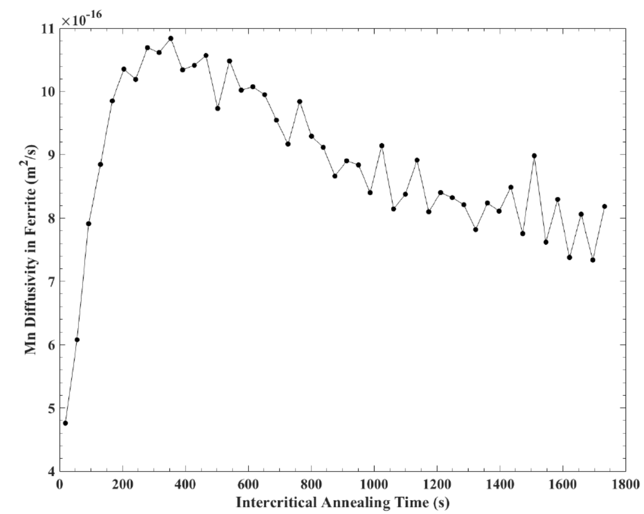
$$D_{Mn\alpha} = \frac{{dx}/{dt}\, 2X\,(C_{Mn\gamma} - C_{Mn0})(C_{Mn\gamma} - C_{Mn\alpha})^2}{(C_{Mn0} - C_{Mn\alpha})^2}$$



AVF vs IA time

- AVF: f_γ=X/210
- Interface velocity at time t :

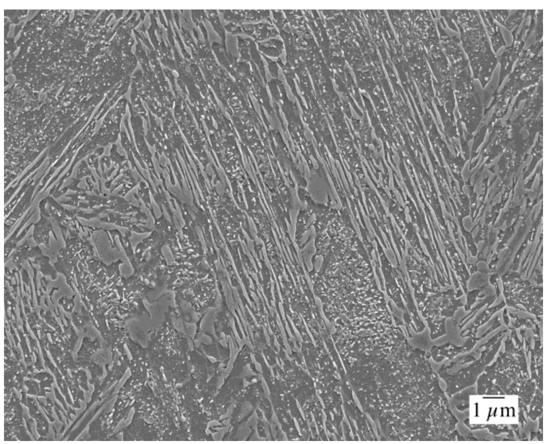
$$ {dx}/{dt} = \frac{X_2 - X_1}{t_2 - t_1} $$



Mn diffusivity in ferrite*
D_0=0.7560x10^{-4}m^2/s, Q=224500J/mol
At 650°C : 1.45X10^{-17}m^2/s
*De Cooman and Speer
Fundamentals of Steel Product Physical Metallurgy

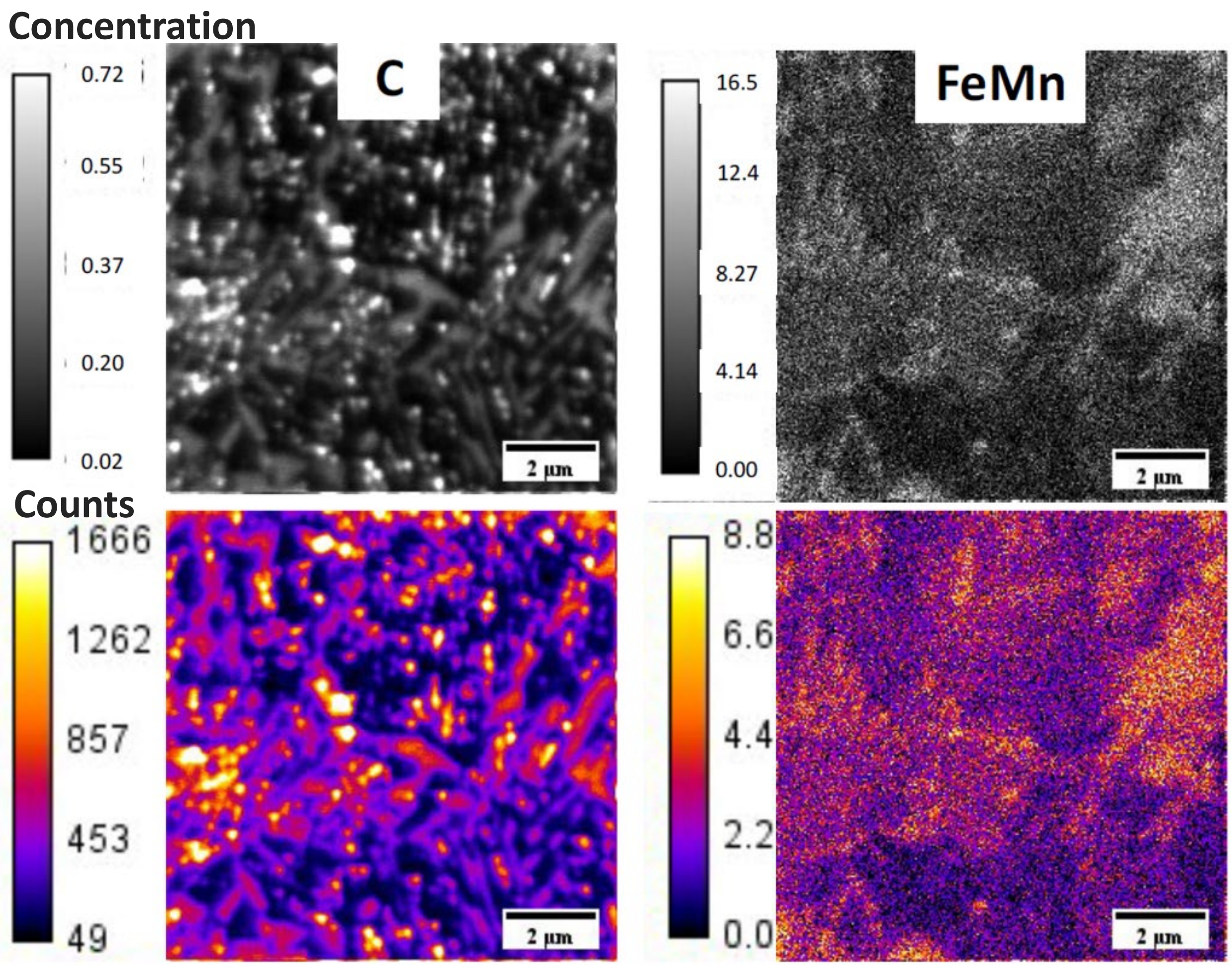
Nano-SIMS experiment to detect C and Mn contents

- 120nm beam
- Carbons were detected with good contrast between the phases whereas Mn were hardly detected due to the limitation of the adopted beam.
- Resolution is not high enough to detect the content gradient near phase boundary.
- TEM, APT may be considered as an alternative.



Material structure with similar IA

0.19C-4.39Mn, 0.8°C/s HR, 650°C & 1800s IA



Nano-SIMS results

Accomplishments to Date

Set-up of Phase Field Simulation Method using MICRESS

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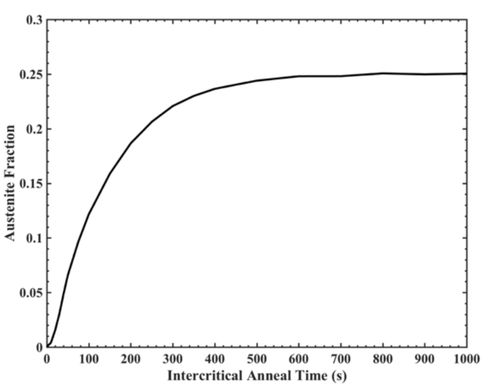
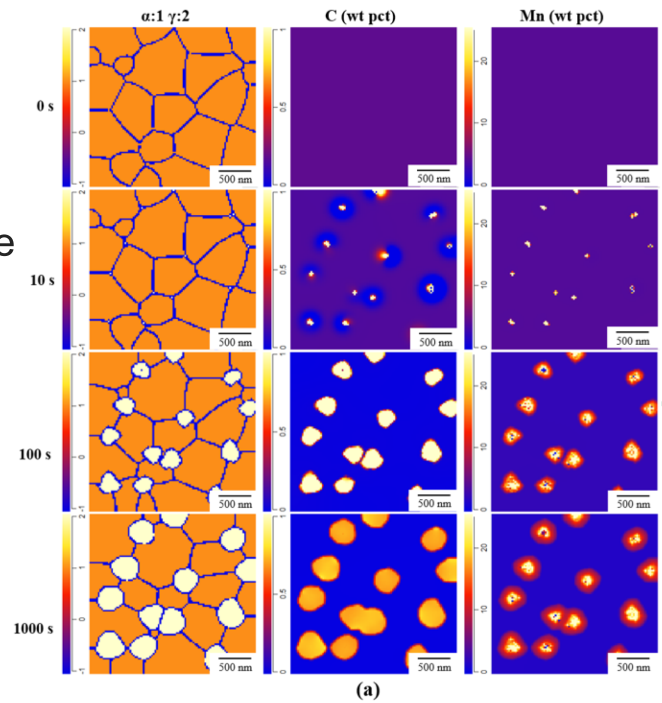
- Amounts of austenite nucleation seeds and their behavior have substantial effects on the austenite growth rate and microstructure evolutions



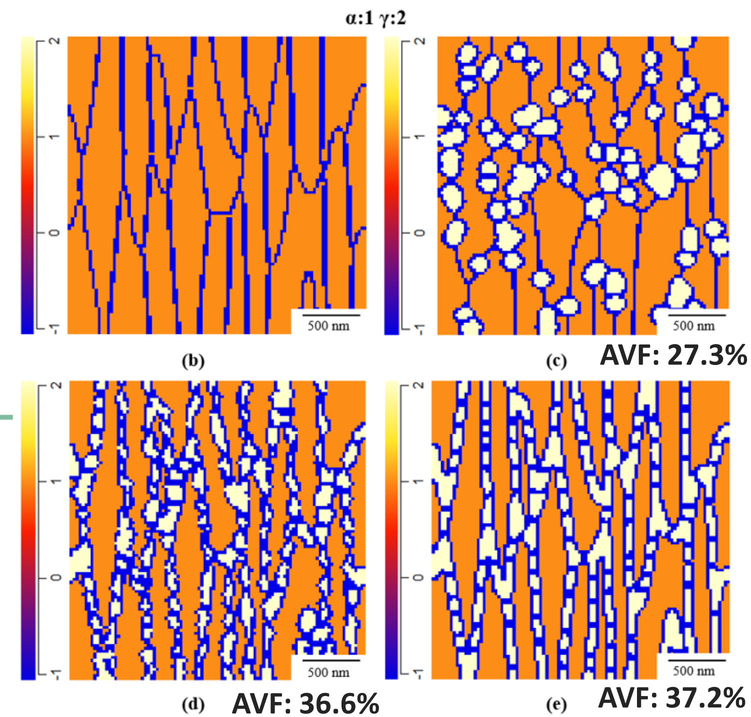
AVF vs. time for the case of equiaxed ferrite

Equiaxed ferrite



Microstructure evolution and C/Mn partitioning vs. time

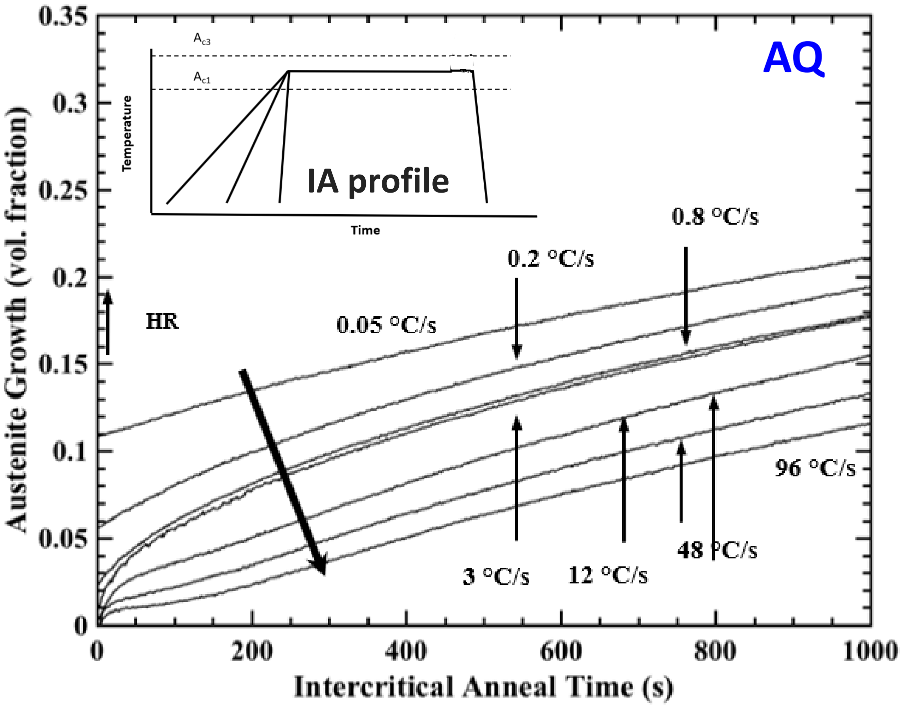
Elongated ferrite



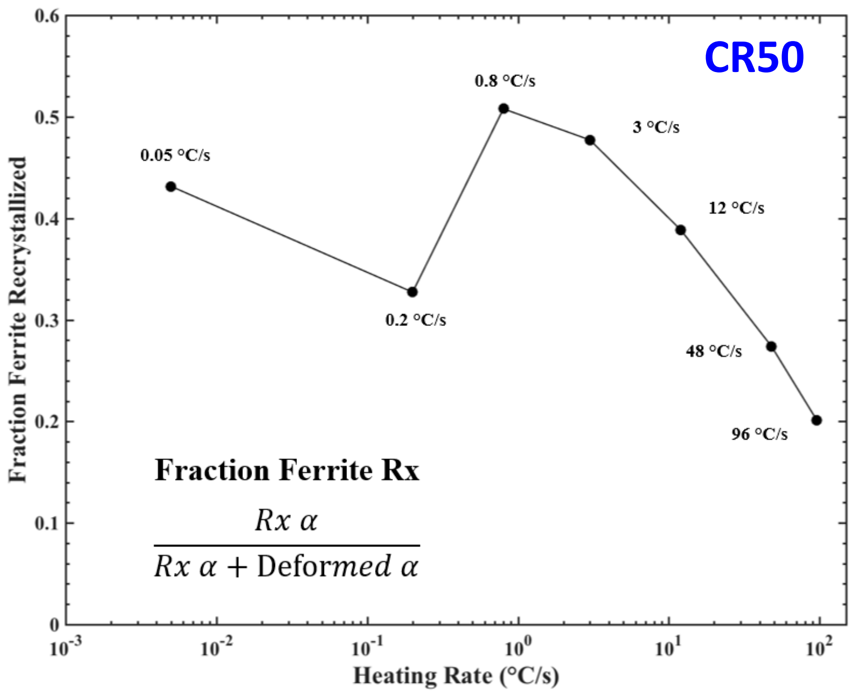
Microstructure evolution after 200s (Effects of amounts of austenite nucleation seeds)

Effects of Heating Rate on Austenite Growth and Ferrite Recrystallization during IA

- 0.0005C-7.19Mn-0.25Si
- CR50: as-received condition with 1.42mm thickness
- AQ: austenitized at 850°C for 300s and water-quenched



In-situ austenite volume fraction from Dilatometry



Ferrite Recrystallization upon Heating

Accomplishments to Date
Ferrite Recrystallization upon Heating

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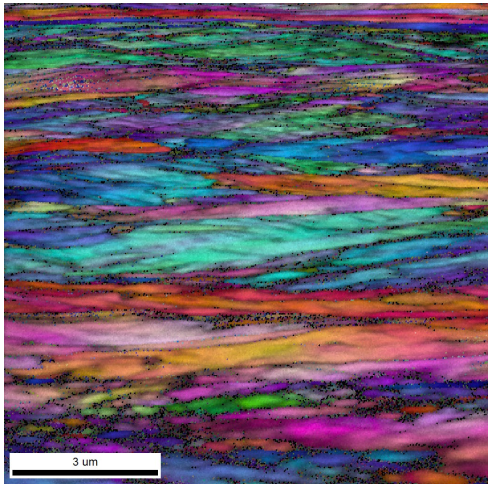
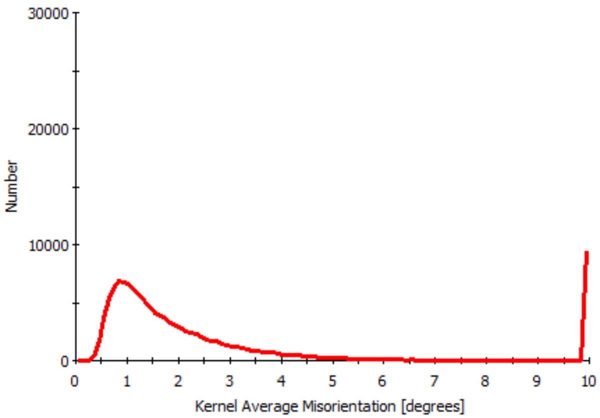
IQ + IPF

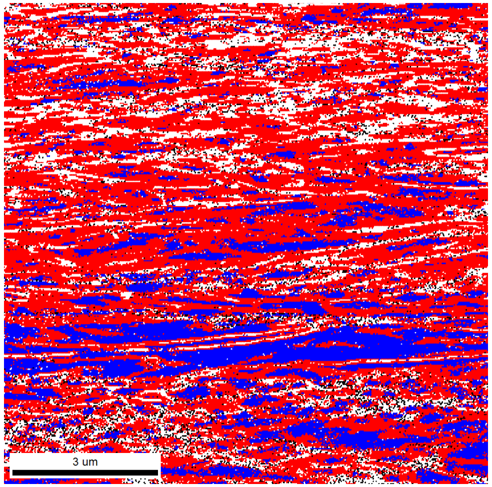
KAM

KAM
Blue: Rx α (0 - 0.9°)
Red: Deformed α (0.9 - 3°)

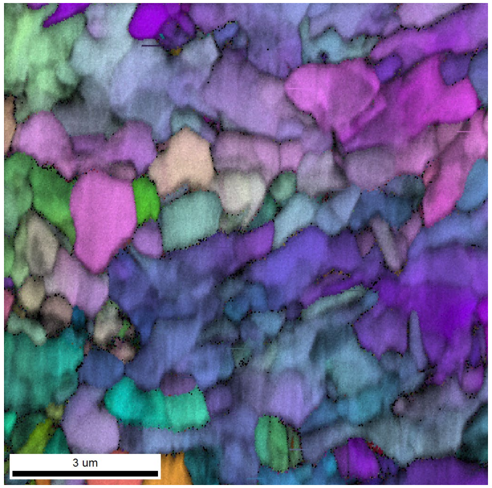
CR50
AR

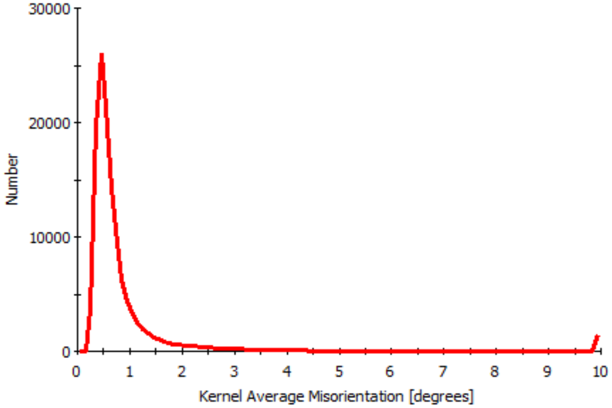


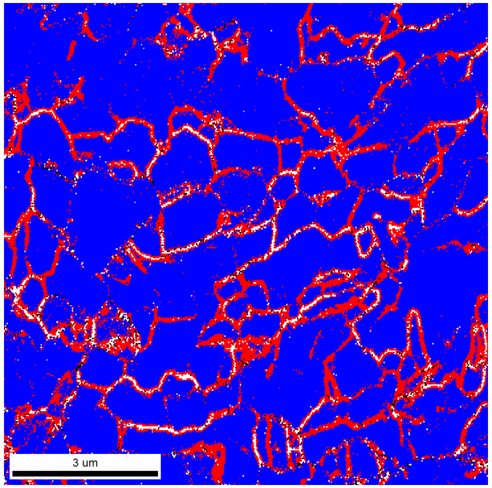




CR50
IA
650 °C
50,000 s







PF simulations for Ferrite Recrystallization and Austenite Growth for Different HR

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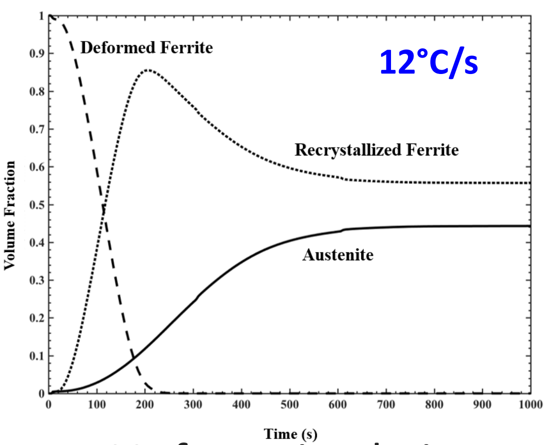
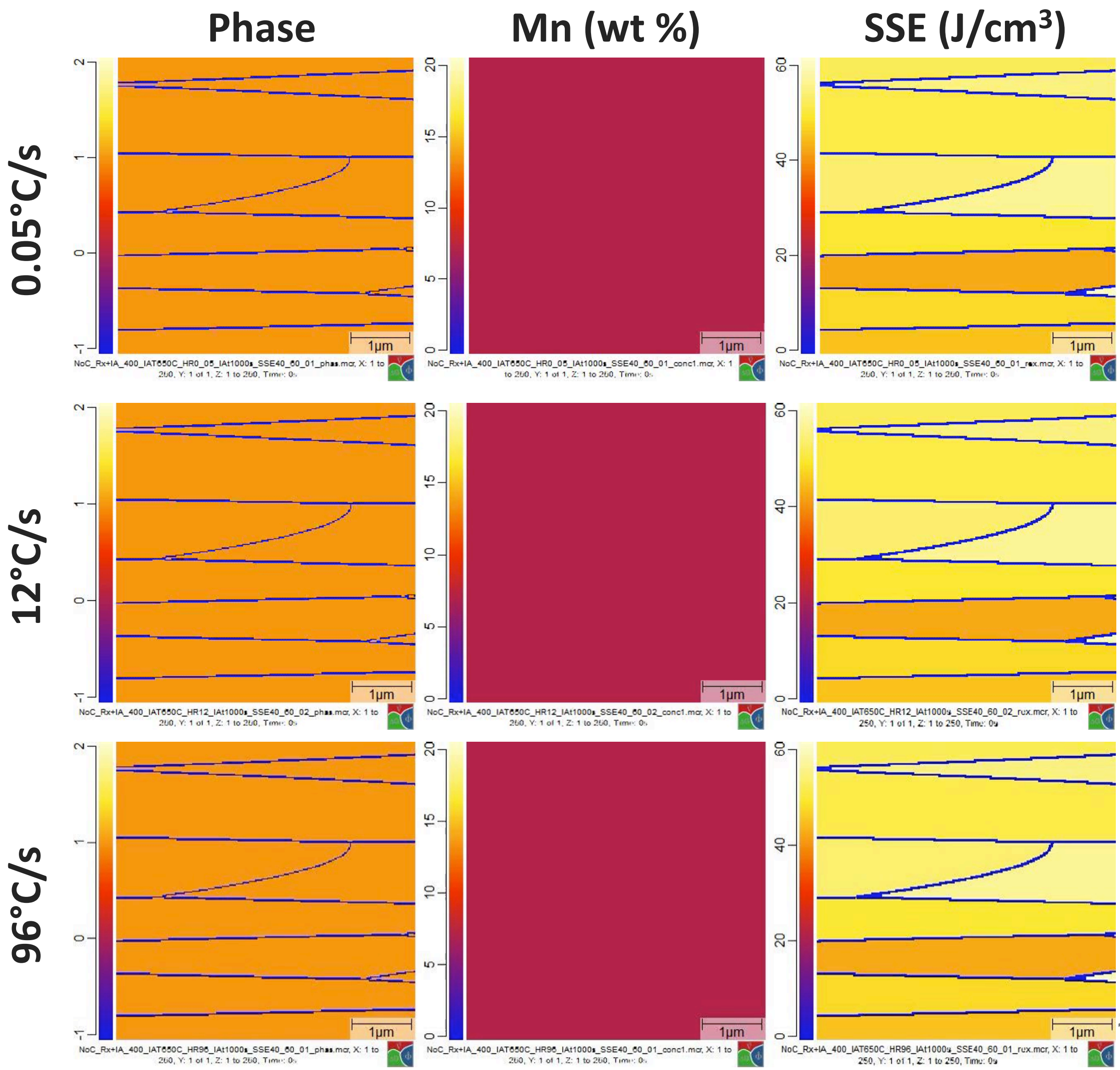
- 7Mn-0.25Si
- 650°C, 1000s IA
- Different microstructure evolutions
- Developed simulation method incorporating HR and ferrite recrystallization



FVF from simulation



Responses to Previous Years Reviewers' Comments

- Comment: Challenging to follow the approach of the work.
  - Response: The description of the approach being taken by the project has been refined in this presentation.
- Comment: Work scope may be taking on too many things at the same time.
  - Response: This project calls for experiments to support simulations while simulations simultaneously feeding back to experiments defining additional measurements. The project tasks communicate with each other to reevaluate the requirements as progress is made.
- Comment: Provide more detail of how the model/framework will be validated.
  - Response: Additional detail is provided in this presentation.
- Comment: How will results be transferred to industry
  - Response: Results and discussion of the framework and approach will be published to make them available to members of industry to access. ASPPRC will be utilized to share and demonstrate progress to industry collaborators.

Collaboration and Coordination

- Computational Engineering Group (CE), Pacific Northwest National Laboratory (PNNL)
  - High throughput In-situ HEXRD heat treatment experiments and characterization
  - Mechanism/microstructure-based finite element modeling of the TRIP assisted 3G AHSS steel with the aid of in-situ HEXRD tensile tests.
- Advanced Steels Processing and Products Center (ASPPRC), Colorado School of Mines (CSM)
  - Ex-situ heat-treatment experiments & materials characterization
  - Microstructural modeling during heat treatment process
- Advanced Photon Source (APS), Argonne National Laboratory (ANL)
  - In-situ HEXRD beamline user facility and in-situ HEXRD experiments assistance.
- Energy and Transportation Science Division, Oak Ridge National Laboratory (ORNL)
  - Advisory

Remaining Challenges and Barriers

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- Obtaining accurate chemical compositions from phase lattice parameters (PLP).
  - Lack of accurate models accounting for both temperature & composition impacts.
- Validation of the phase field microstructure/phase transformation modeling of the whole heat treatment processes of phase transformation and C, Mn partitioning.
  - Nano-SIMS measurements provided data for C partitioning. Exploration of measurement techniques of Mn partitioning is ongoing
- Accurate mechanism/microstructure-based modeling of Med-Mn 3G AHSS.
  - We have achieved initial success on qualitatively capturing Luders band behavior by the consideration of Bain strain. More accurate models (including kinematic hardening) and comprehensive studies are needed.

Proposed Future Work

- Continue the study on correlating phase lattice parameter changes with chemical composition change during IA heat treatment process.
- Continue the development of a phase field model which will be able to used for
  - Determine an accurate measurement technique for Mn partitioning for model validation.
- Continue the development of mechanism/microstructure-based modeling of Med-Mn 3G AHSS mechanical performance during forming.
- Conduct addition in-situ HEXRD tensile tests to provide validation data for the phase field and mechanism/microstructure-based models.
- Using the information of the correlation between microstructure and mechanical performance, determine optimized IA parameters to obtain desired microstructures using TCD/phase field based modeling.

Any proposed future work is subject to change based on funding levels

Summary

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- SEM-EDAX microstructural and composition analysis has been performed on as-received 7Mn and 10Mn steels to obtain information of Mn, Al, Si partitioning in austenite and ferrite phases.
- Ex-Situ heat treatment and characterization (SEM-EDAX) has been performed on 5Mn steels.
- High throughput *in-situ* HEXRD measurement technique has been developed & used to obtain AVS and PLP during inter-critical annealing (IA) cycles of 5Mn (as-quenched), 7Mn and 10Mn (as-received) steels.
- ThermoCalc-Dictra (TCD) simulations for 5Mn steels (as-quenched) haven been performed to study martensitic transformation and C, Mn partitioning during IA holding.
- The Mn mobility in Ferrite/martensite in 5Mn steel has been determined by integrated HEXRD AVS measurements and TCD microstructure modeling.
- Phase field modeling has been conducted to studying microstructure evolution and partitioning over time as well as the effects of heating rate.
- Non incremental *In-situ* HEXRD tensile tests of 7Mn & 10Mn steels along different directions to better understand the TRIP effect and the yield point elongations (Luders band behavior) of medium Mn steels.
- Phenomenological and Crystal plasticity FE models have been established with the consideration of martensitic transformation and the Luders band and yield point elongation (YPE) has been successfully captured by the models.